SULFIDE BY METHYLENE BLUE METHOD **SM 4500-S2 D – 2000 (2011)**

ADDITIONAL QC REQUIREMENTS FOR THIS METHOD: Certified or Accredited laboratories using this method are assessed to applicable requirements of SM 1020 and SM 4020.

Facility Name:_____VELAP ID_____

Assessor Name:_____Analyst Name:_____Inspection Date_____

Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Records Examined: SOP Number/ Revision/ Date _____ Analyst:_____ Sample ID: _____ Date of Sample Preparation:_____ Date of Analysis:_____					
1) Were Non-Potable Water samples preserved with zinc acetate plus sodium hydroxide to pH>9; stored at ≤6°C; and held for not longer than 7 days?	40 CFR 136 Table 1I				
2) Were solid samples and sediments stored at 4°C or frozen and analyzed within 2 weeks (or 1 month for frozen samples)?	4500-S^{2-} A.3				
3) If preparing sulfide stock in-house, was solution prepared from sodium sulfide nonahydrate ($Na_2S \cdot 9H_2O$) crystals?	4500-S^{2-} A.6				
4) If preparing sulfide stock in-house, were $Na_2S \cdot 9H_2O$ crystals kept for no longer than 1 year?	4500-S^{2-} A.6				
5) If preparing sulfide stock in-house, was reagent water used in standard preparation degassed with argon or nitrogen?	4500-S^{2-} A.6				
6) If preparing stock in-house, was it standardized using the iodometric method, 4500-S2- F? *(Alternatively, purchase precertified stock standard.)*	4500-S^{2-} A.6				
7) Was the concentration of stock solution verified daily using the iodometric method (F)?	4500-S^{2-} A.6				
8) Are samples that are collected for the determination of dissolved sulfides treated in the field with aluminum chloride, allowed to settle and flocculate, and the clear supernatant collected for analysis of dissolved sulfide? *(Step may be omitted if sample contains no suspended matter.)*	4500-S^{2-} B				
Notes/Comments:					

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9) When samples are pre-treated in the field for the determination of dissolved sulfides, does the chain of custody or other documentation include a record of flocculation step?	2003 NELAC 5.5.7.3				
10) Was the Methylene Blue Solution I standardized against five known concentration sulfide standards ranging from 1 to 8 mg Sulfide/L?	4500-S^{2-} D 2.f				
11) Was Methylene Blue Solution I prepared fresh daily?	4500-S^{2-} D 2.f				
12) Was Methylene Blue Solution II prepared by diluting 10 mL of Methylene Blue Solution I to 100 mL with reagent water?	4500-S^{2-} D 2.g				
13) Were 7.5 aliquots of sample analyzed in two matched tube sets?	4500-S^{2-} D 3.a				
14) Were 0.5 mL amine-sulfuric acid reagent and 0.15 mL (3 drops) $FeCl_3$ added to the first tube and mixed slowly by inverting only once? *("Excessive mixing causes low results by loss of H_2S as a gas before it has had time to react.")*	4500-S^{2-} D 3.a				
15) Were 0.5 mL 1:1 H_2SO_4 and 0.15 mL FeCl3 Solution added to the second tube and mixed?	4500-S^{2-} D 3.a				
16) After 3-5 minutes of color, was 1.6 mL $(NH_4)_2HPO_4$ added to each tube?	4500-S^{2-} D 3.a				
17) Were color comparisons made after waiting 3-15 minutes?	4500-S^{2-} D 3.a				
18) If using visual color determination, was Methylene Blue solution I or II added (depending on sulfide concentration and desired accuracy) dropwise to the second tube until color matches that developed in the first tube?	4500-S^{2-} D 3.b.1				
19) For visual determination, if the concentration exceeds 20 mg/L, is the test repeated with sample diluted tenfold?	4500-S^{2-} D 3.b.1				
20) For visual determination, are results calculated per the following? "With methylene blue solution I, adjusted so 0.05 mL (1 drop) = 1.0 mg S^{2-}/L when 7.5 mL of sample are used: mg S^{2-}/L = no. drops solution I + 0.1 (no. drops solution II)"	4500-S^{2-} D 3.b.1				
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21) For photometric color determination, is the instrument zeroed with treated sample from the second tube?	4500-S^{2-} D 3.b.2				
22) For photometric determination, is sample concentration determined from calibration curve?	4500-S^{2-} D 3.b.2				
23) When samples are pre-treated in the field (flocculation), is the analysis result reported as Dissolved Sulfide?	2003 NELAC 5.5.10.2.h				

Notes/Comments: